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(54) Title: METHOD OF USING AN ARALKYLSILOXANE			
(57) Abstract			
<p>A method of using of an aralkyalkylsiloxane includes adding the aralkylsiloxane as a component in a personal care composition, in an amount effective to enhance one or more properties, such as shine, lubricity and visual masking of inorganic components, of the personal care composition.</p>			

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METHOD OF USING AN ARALKYLSILOXANE

FIELD OF THE INVENTION

The invention relates to the use of certain siloxane compounds, more particularly to the use of certain aralkylsiloxane compounds.

BRIEF DESCRIPTION OF THE RELATED ART

The use of phenyltrimethicone in personal care compositions, for example, skin lotions, antiperspirants, hair care products to enhance shine, lubricity and/or visual masking of inorganic components, is known. Typical methods for making phenyltrimethicone, such as for example, by cohydrolysis of phenyltrichlorosilane and chlorotrimethylsilane may introduce impurities, such as for example, side products and catalyst residues, into the phenyltrimethicone product which are undesirable in the context of using the phenyltrimethicone product as a component in a personal care composition.

SUMMARY OF THE INVENTION

In a first aspect, the present invention is directed to a method of using of an aralkylsiloxane, comprising adding the aralkylsiloxane as a component in a personal care composition, in an amount effective to enhance one or more properties of the personal care composition. For example, the aralkylsiloxane enhances the shine, emolliency and lubricity of personal care compositions and aids in visually masking inorganic components of such compositions.

In a second aspect, the present invention is directed to a method for making an aralkyl siloxane, comprising contacting a silylhydride-functional polysiloxane with a terminally unsaturated arylalkene under hydrosilylation conditions. Aralkylsiloxanes made by the preferred process exhibit a low, preferably an undetectably low, level of impurities and thus allow the amount

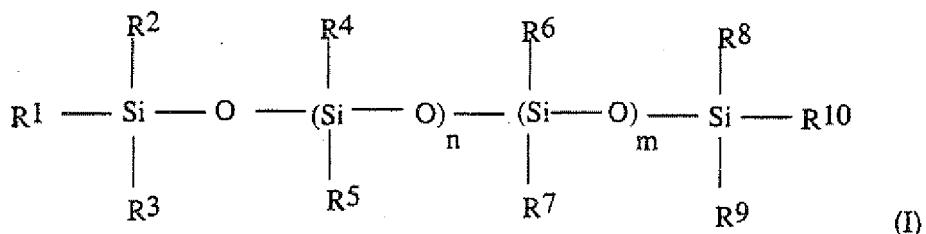
of impurities introduced into the personal care composition by addition of such aralkylsiloxanes to be minimized.

In a third aspect, the present invention is directed to a method for making an improved personal care composition, said personal care composition being an analog of a first personal care composition that contains phenyltrimethicone as a component thereof, comprising making a personal care composition analogous to the first personal care composition wherein an aralkylsiloxane is substituted for the phenyltrimethicone of the first personal care composition.

DETAILED DESCRIPTION OF THE INVENTION

In a preferred embodiment, the personal care composition comprises, based on 100 parts by weight ("pbw") of the personal care composition, from about 0.5 pbw to about 50 pbw, more preferably from about 1 pbw to about 30 pbw, still more preferably from about 2 pbw to about 20 pbw, of the aralkylalkylsiloxane.

Aralkylsiloxanes that are suitable as the aralkylsiloxane component of the present invention are those comprising one or more compounds according to formula (I):



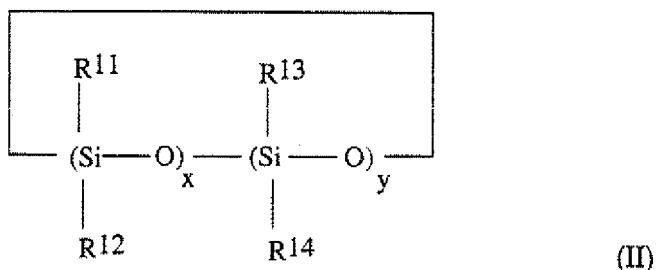
wherein:

R¹, R², R³, R⁴, R⁵, R⁸ and R⁹ and R¹⁰ are each independently H, alkyl, more preferably (C₁-C₆)alkyl, haloalkyl, more preferably halo(C₁-C₆)alkyl, aryl or aralkyl,

R⁶, R⁷ are each independently H, alkyl, more preferably (C₁-C₆)alkyl, 5 haloalkyl, more preferably halo(C₁-C₆)alkyl, or aryl;

n and m are each independently integers from 0 to 6, provided that (n + m) \geq 1, preferably 1 \leq (n+m) \leq 8, still more preferably 1 \leq (n+m) \leq 4 and provided that at least one substituent group of the compound of formula (I) is aralkyl;

10 or of formula (II):



wherein:

R¹¹ and R¹² are each independently H, alkyl, more preferably (C₁-C₆)alkyl, haloalkyl, more preferably halo(C₁-C₆)alkyl, aryl or aralkyl;

15 R¹³ and R¹⁴ are each independently H, alkyl, more preferably (C₁-C₆)alkyl, haloalkyl, more preferably halo(C₁-C₆)alkyl, or aryl;

x and y are each independently integers from 0 to 6, provided that 3 \leq (x + y) \leq 10, preferably 4 \leq (x + y) \leq 6, and provided that at least one substituent group of the compound of formula (II) is aralkyl.

As used herein, the term "(C₁-C₆)alkyl" means a linear or branched alkyl group containing from 1 to 6 carbons per group, such as, for example, methyl, ethyl, propyl, iso-propyl, n-butyl, iso-butyl, sec-butyl, tert-butyl, pentyl, hexyl, preferably methyl.

5 As used herein, the term "halo(C₁-C₆)alkyl" means a linear or branched alkyl group containing from 1 to 6 carbons per group that is substituted with one or more halo substituents, such as, for example, chloromethyl, trifluoromethyl.

10 As used herein, the term "aryl" means a monovalent unsaturated hydrocarbon ring system containing one or more aromatic rings per group, which may optionally be substituted on the one or more aromatic rings, preferably with one or more groups selected from amino, nitro, (C₁-C₆)alkyl, and which, in the case of two or more rings, may be fused rings, including, for example, phenyl, 2,4,6-trimethylphenyl, 2-isopropylmethylphenyl, 1-15 pentenyl, naphthyl, anthryl, preferably phenyl.

20 As used herein, the term "aralkyl" means an aryl derivative of an alkyl group, preferably a (C₂-C₆)alkyl group, wherein the alkyl portion of the aryl derivative may, optionally, be interrupted by an oxygen atom, such as, for example, phenylethyl, phenylpropyl, 2-(1-naphthyl)ethyl, preferably phenylpropyl, phenoxypropyl, biphenyloxypropyl.

In a highly preferred embodiment, the aralkylsiloxane comprises a compound according to formula (I), wherein R¹ and R¹⁰ are each aralkyl, more preferably phenylpropyl, R², R³, R⁶, R⁷, R⁸ and R⁹ are each (C₁-C₆) alkyl, more preferably methyl, n is 0 and 2 ≤ m ≤ 5, more preferably n is 0 and m is 3. In 25 a very highly preferred embodiment, the aralkylsiloxane comprises α,ω-bis(2-phenylpropyl)siloxane.

In a preferred embodiment, the aralkylsiloxane comprises a compound according to formula (I), wherein R¹, R², R³, R⁴, R⁶, R⁷, R⁸, R⁹ and R¹⁰ are each (C₁-C₆) alkyl, preferably methyl, R⁵ is aralkyl, more preferably phenylpropyl, n is 1 or 2 and 1 ≤ m ≤ 8, more preferably, n is 1 and m is 2.

5 In a preferred embodiment, the aralkyl siloxane comprises a compound according to the structural formula (II), wherein R¹¹, R¹³ and R¹⁴ are each (C₁-C₆) alkyl, preferably methyl, R¹² is aralkyl, more preferably phenylpropyl, 1 ≤ x ≤ 4 and 2 ≤ y ≤ 10, more preferably x is 1 and y is 4.

10 In a highly preferred embodiment, the aralkyl content of the aralkylsiloxane is selected to provide a refractive index of from 1.40 to 1.50, more preferably from about 1.44 to 1.48, at 25°C. In a highly preferred embodiment, the aralkylsiloxane comprises, based on the molecular weight of the aralkylsiloxane, from about 25 to about 65 percent by weight ("wt%"), more preferably from about 30 to about 50 wt% and still more preferably from 15 about 33 to about 45 wt%, of one or more aralkyl substituent groups.

Suitable aralkylsiloxane can be made by known methods, see, for example, U.S. Patent No. 3,088,964 and 5,300,669 and 5,384,383, the disclosures of which are each incorporated herein by reference.

20 In a preferred embodiment, the aralkylsiloxane is made by contacting a silylhydride-functional polysiloxane with an terminally unsaturated arylalkene, such as for example, styrene or 2-phenylpropene, under hydrosilylation conditions, preferably in the presence of a solid hydrosilylation catalyst. In a preferred embodiment, the solid hydrosilylation catalyst comprises a catalytically active metal, such as for 25 example, platinum, supported on an inert support, such as for example, alumina. Preferably, the aralkylsiloxane product exhibits a hydride content of less than 100 parts per million ("ppm"), based on the weight of

aralkylsiloxane. The catalyst and excess arylalkene may be easily removed from the product mixture to yield an aralkylsiloxane having a low to undetectably low content of impurities. In a preferred embodiment, the solid catalyst is removed from the aralkylsiloxane product by filtration. Excess 5 arylalkene may be removed by heating the product mixture by vacuum stripping the aralkylsiloxane.

The personal care applications in which an aralkylalkylsiloxane may be employed include, but are not limited to, deodorants, antiperspirants, skin lotions, moisturizers, hair care products such as shampoos, hair conditioners, 10 mousses, hair sprays and styling gels, protective compositions such as sunscreens, skin treatments and anti-aging products, color cosmetics such as lipsticks, foundations, blushes, makeup, and mascaras; and other cosmetic formulations, as well as drug delivery systems for topical application of medicinal compositions that are to be applied to the skin. Suitable personal 15 care compositions are made by combining, according to methods known in the art, one or more of the above components with an aralkylalkylsiloxane.

In a preferred embodiment, suitable for example, as an after bath oil composition, the personal care composition of the present invention comprises a mineral oil and an aralkylsiloxane.

20 In a preferred embodiment, suitable for example, as a hair conditioner composition, the personal care composition of the present invention comprises a suitable solvent, such as for example, isododecane, isohexadecane or cyclomethicone and an aralkylsiloxane.

25 In a preferred embodiment, suitable for example, as a skin care composition, the personal care composition of the present invention comprises the aralkylsiloxane and a nonaqueous medium, comprising for example one or more of organic oils, such as sunflower seed oil and

octyldodecylneopentanoate, and silicone oils, such as dimethicone and cyclomethicone, or with an aqueous medium, preferably in the form of an emulsion. Preferably the skin care composition further comprises one or more suitable emulsifiers, such as, for example, fatty acid esters, sorbitan derivatives such as sorbitan sesquioleate, sorbitan oleate, sorbitan isostearate, 5 polyglyceryl-3 oleate, alkoxylated alcohols such as laureth-4, laureth-7, deceth-12, steareth-10, hydroxylated or alkoxylated derivatives of silicone compounds such as dimethicone copolyol, cetyl dimethicone copolyol, and lauryl methicone copolyol and glyceryl esters such as polyglyceryl-4-10 isostearyl.

In a preferred embodiment, suitable as an anti-perspirant composition the personal care composition of the present invention comprises one or more active anti-perspirant agents, such as, for example, aluminum halides, aluminum hydroxyhalides, for example, aluminum chlorohydrate, and 15 complexes or mixtures thereof with zirconyl oxyhalides and zirconyl hydroxyhalides, such as for example, aluminum-zirconium chlorohydrate, and an aralkylsiloxane.

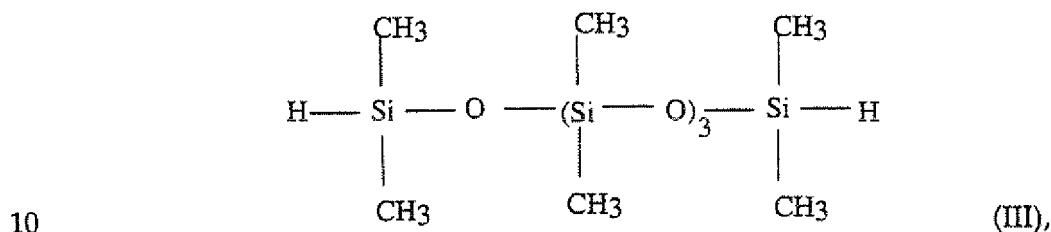
In a preferred embodiment, suitable, for example, as a sunscreen composition, the personal care composition of the present invention comprises one or more absorbing or blocking agents for ultraviolet radiation, 20 such as, for example, titanium dioxide, p-aminobenzoic acid and oxybenzone sunscreens, such as benzophenone-3 and an aralkylsiloxane. In a preferred embodiment, the sunscreen composition is in the form of an aqueous emulsion, further comprising water and one or more emulsifiers.

25 The personal care composition of the present invention may, optionally, further contain such known components as, for example, emollients, moisturizers, humectants, pigments, colorants, fragrances, biocides, preservatives, exfoliants, hormones, enzymes, medicinal

compounds, anti-microbial agents, anti-fungal agents, vitamins, salts, electrolytes, alcohols, polyols, absorbing or blocking agents for ultraviolet radiation, botanical extracts, surfactants, emulsifiers, silicone oils, organic oils, waxes, thickening agents such as, for example, acrylic polymers, fumed silica or hydrated silica, clays, such as, for example, bentonite, and organo-modified clays.

Example 1

A reaction vessel was charged with 450 pbw of an equilibrated hydride-terminated siloxane oligomer having an average structure according to formula (III):



10

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0.15 pbw of 5wt % platinum on alumina. The reaction mixture was heated to 130°C at which point the addition of 292 pbw α -methyl styrene was begun. The progress of the reaction was followed by gasiometric analysis of the remaining hydride. After 2 hours at 130°C, the hydride level had been reduced to 62 parts per million. Excess α -methyl styrene was then removed by heating the contents of the reaction vessel to 150°C under reduced pressure with a nitrogen sparge and the catalyst was removed by filtration to yield a clear, colorless liquid having a refractive index of 1.4650 at 25°C.

Example 2 and Comparative Examples C1 and C2

The solid stick antiperspirant compositions of Example 2 and Comparative Examples C1 and C2 were made by combining the components in the relative amounts, expressed in pbw, set forth below in Table I.

Each of the compositions was made by: (i) mixing the siloxane component(s), that is, the cyclopentasiloxane and, as applicable, the phenyl trimethicone or α,ω -bis(2-phenylpropyl)siloxane, with the stearyl alcohol component, (ii) adding the aluminum zirconium chlohydex gly, talc and glycetyl stearate and PEG-100 stearate components, (iii) heating the mixture of components to 75°C and stirring with moderate agitation until the waxes are melted, (iv) adding melted hydrogenated castor oil to the mixture and stirring for 15 minutes, and (v) cooling the mixture to 55°C and pouring the mixture into containers.

Each of the compositions was applied to skin and evaluated for the amount of white residue visible on the skin after application. The results of the evaluation are indicated in TABLE 1, with a "+" indicating that white residue was visible and a "-" indicating that no white residue was visible. Neither Example 2 nor Comparative Example C2 left a visible white residue.

TABLE 1

	2	C1	C2
Cyclopentasiloxane	40	50	40
Phenyl trimethicone	--	--	10
α,ω -bis(2-phenylpropyl)siloxane	10	--	--
Stearyl alcohol	19	19	19
Hydrogenated castor oil	3	3	3
Talc	4	4	4
Glyceryl stearate and PEG-100 stearate	2	2	2
Aluminum zirconium chlohydex gly	22	22	22
Appearance on skin,			
white residue visible (+)			
no white residue visible (-)	-	+	-

Example 3 and Comparative Examples C3 and C4

The skin lotion compositions of Example 3 and Comparative Examples C3 and C4 were made by combining the components in the relative amounts, expressed in pbw, set forth below in Table II.

5 The compositions were made by (i) combining the components to from Part B and (ii) heating Part B to 70°C, (iii) combining the components to form Part A and heating Part A until melted, (iv) slowly adding molten Part A to Part B, (v) mixing the combined parts A and B for 30 minutes and (vi) cooling the mixture with continued mixing.

TABLE II

	3	C3	C4
Part A			
Phenyltrimethicone	—	—	3.6
α,ω-bis(2-phenylpropyl)siloxane	3.6	—	—
stearic acid	1.4	1.4	1.4
cetyl alcohol	1.0	1.0	1.0
Part B			
Glycerin	2.0	2.0	2.0
Triethanolamine	0.8	0.8	0.8
Methyl paraben	0.15	0.15	0.15
Deionized water	91.05	94.65	91.05

10 The compositions of Example 3 and Comparative Examples C3 and C4 were applied to skin. Compared to the composition of Comparative Example C3, the compositions of Example 3 and Comparative Example C4 each felt less greasy and tacky during application, were rubbed into the skin faster and provided a more lubricious feel after drying.

Example 4 and Comparative Examples C5 and C6

The after bath oil compositions of Example 4 and Comparative Examples C5 and C6 were made by combining the components in the relative amounts, expressed in pbw, set forth below in Table III. The silicone components were combined and mixed until homogeneous, the mineral oil was then added to the combined silicone components while mixing and mixing of the combined silicone components and mineral oil was continued for 15 minutes.

TABLE III

	4	C5	C6
Phenyltrimethicone	--	--	10
α,ω -bis(2-phenylpropyl)siloxane	10	--	--
cyclopentasiloxane	58	58	58
light mineral oil	32	42	32

The after bath oil compositions of Example 4 and Comparative Examples C5 and C6 were each applied to skin. The composition of Comparative Example C5 dried quickly and did not provide a lubricious feel to the skin after drying. The composition of Comparative Example C6 took slightly longer to dry than did the composition of Comparative Example C5 and provided a soft, smooth and lubricious, but somewhat greasy feel to the skin after drying. The composition of Example 4 took slightly longer to dry than did the composition of Comparative Example C5 and provided a soft smooth and lubricious feel, which was less greasy than that provided by the composition of Comparative Example C6 to the skin after drying.

Example 5 and Comparative Examples C7 and C8

The hair cuticle coating compositions of Example 5 and Comparative Examples C7 and C8 were made by combining the components in the relative amounts, expressed in pbw, set forth below in Table IV. The cyclomethicone, dimethicone and isohexadecane components were combined and the α,ω -bis(2-phenylpropyl)siloxane or phenyltrimethicone was then added to the combined cyclomethicone, dimethicone and isohexadecane components with mixing and the mixing was continued for 15 minutes.

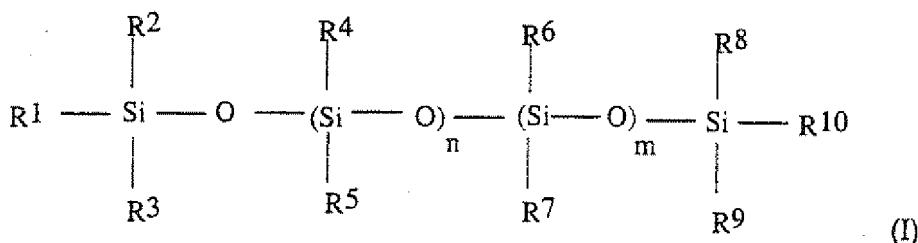
TABLE IV

	5	C7	C8
Cyclomethicone and dimethicone	60	60	60
Phenyltrimethicone	--	--	30
α,ω -bis(2-phenylpropyl)siloxane	30	--	--
isohexadecane	10	40	10

The compositions of Example 5 and Comparative Examples C7 and C8 were applied to hair tresses. Each of the compositions initially provided shine and conditioning properties to the hair tresses. Upon drying, the tresses treated with composition of Comparative Example C7 exhibited a dull appearance, while those treated with the compositions of Example 5 and Comparative Example C8 continued to exhibit a shiny appearance.

WHAT IS CLAIMED IS:

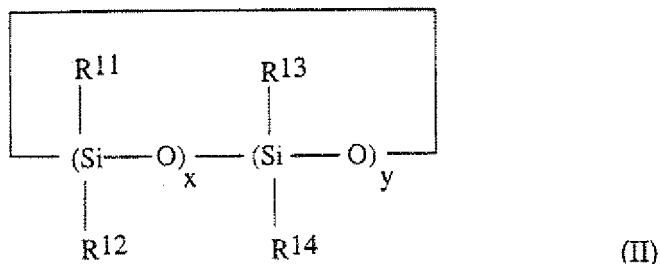
1. A method of using of an aralkylsiloxane, comprising adding the aralkylsiloxane as a component in a personal care composition, in an amount effective to enhance one or more properties of the personal care composition.
2. The method of claim 1, wherein the aralkyl siloxane is made by contacting a silylhydride-functional polysiloxane with a terminally unsaturated arylalkene under hydrosilylation conditions.
3. The method of claim 2, wherein the silylhydride-functional polysiloxane and terminally unsaturated arylalkene are contacted in the presence of a supported catalyst and wherein the supported catalyst is removed from the aralkylsiloxane by filtration.
4. The method of claim 1, wherein the aralkylsiloxane comprises one or more compounds according to formula (I)



wherein:

- 15 R^1 , R^2 , R^3 , R^4 , R^5 , R^8 and R^9 and R^{10} are each independently H, alkyl, haloalkyl, aryl or aralkyl;
- 20 R^6 , R^7 are each independently H, alkyl, haloalkyl or aryl;
- n and m are each independently integers from 0 to 6, provided that $(n + m) \geq 1$ and provided that at least one substituent group of the compound of formula (I) is aralkyl;

or of formula (II):



wherein:

R¹¹ and R¹² are each independently H, alkyl, haloalkyl, aryl or aralkyl;

5 R¹³ and R¹⁴ are each independently H, alkyl, haloalkyl or aryl;

x and y are each independently integers from 0 to 6, provided that 3 ≤ (x + y) ≤ 10 and provided that at least one substituent group of the compound of formula (II) is aralkyl.

10 5. The method of claim 4, wherein the aralkylsiloxane comprises a compound according to formula (I), R¹ and R¹⁰ are each aralkyl, R², R³, R⁶, R⁷, R⁸ and R⁹ are each (C₁-C₆) alkyl, n is 0 and 2 ≤ m ≤ 5.

6. The method of claim 4, wherein the aralkylsiloxane comprises α,ω-bis(2-phenylpropyl)siloxane.

15 7. The method of claim 4, wherein the aralkylsiloxane comprises a compound according to formula (I), R¹, R², R³, R⁶, R⁷, R⁸, R⁹ and R¹⁰ are each (C₁-C₆) alkyl, R⁴ and R⁵ are each independently each (C₁-C₆) alkyl or aralkyl, provided that at least one of R⁴ and R⁵ is aralkyl, n is 1 or 2 and 1 ≤ m ≤ 8.

20 8. The method of claim 7, wherein R¹, R², R³, R⁴, R⁶, R⁷, R⁸, R⁹ and R¹⁰ are each methyl, R⁵ is phenylpropyl, n is 1 and m is 2.

9. The method of claim 4, wherein the aralkylsiloxane comprises a compound according to the structural formula (II), R11, R13 and R14 are each (C1-C6) alkyl, R12 is aralkyl, $1 \leq x \leq 4$ and $2 \leq y \leq 10$.

10. The method of claim 9, wherein R11, R13 and R14 are each 5 methyl, R12 is phenylpropyl, x is 1 and y is 4.

11. The method of claim 4, wherein the relative amount of aralkyl substituent groups is selected to provide an aralkylsiloxane that exhibits a refractive index of from 1.40 to 1.50 at 25°C.

12. The method of claim 1, wherein the aralkylsiloxane comprises, 10 based on the molecular weight of the aralkylsiloxane, from about 25 to about 65 percent by weight of one or more aralkyl substituent groups.

13. A personal care composition made by the method of claim 1.

14. The personal care composition of claim 13, wherein the 15 composition comprises one or more active anti-perspirant agents and the aralkylsiloxane.

15. The personal care composition of claim 13, wherein the composition comprises water, an emulsifier and the aralkylsiloxane.

16. The personal care composition of claim 13, wherein the 20 composition comprises the aralkylsiloxane and one or more of silicone oils and organic oils.

17. The personal care composition of claim 13, wherein the composition comprises a solvent and the aralkylsiloxane.

18. The personal care composition of claim 13, wherein the 25 composition comprises one or more absorbing agents for ultraviolet radiation and the aralkylsiloxane.

19. A method for making an aralkyl siloxane, comprising contacting a silylhydride-functional polysiloxane with a terminally unsaturated arylalkene under hydrosilylation conditions in the presence of a solid hydrosilylation catalyst and removing the solid hydrosilylation catalyst from 5 the aralkysiloxane by filtration.

20. A method for making an improved personal care composition, said personal care composition being an analog of a first personal care composition that contains phenyltrimethicone as a component thereof, comprising making a personal care composition analogous to the first personal care composition wherein an aralkylsiloxane is substituted for the phenyltrimethicone of the first personal care composition.
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INTERNATIONAL SEARCH REPORT

Int. Application No

PCT/US 99/17746

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A61K7/06 A61K7/48

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the International search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 549 223 A (DOW CORNING) 30 June 1993 (1993-06-30) claims 1,2; examples	1-5, 13-17
X	US 5 160 730 A (DUBIEF CLAUDE ET AL) 3 November 1992 (1992-11-03) claims 1-4; examples	1-5,13
X	EP 0 756 865 A (OREAL) 5 February 1997 (1997-02-05) claims 1-3,15	1-5,13
X	US 5 384 383 A (LEGROW GARY E ET AL) 24 January 1995 (1995-01-24) cited in the application claims 1,12-15	19

 Further documents are listed in the continuation of box C. Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

Information on patent family members

Int'l	Serial Application No
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